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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
09/831,198	05/07/2001	George Douglas Hiler II	7359	1446

27752 7590 06/21/2006

THE PROCTER & GAMBLE COMPANY  
INTELLECTUAL PROPERTY DIVISION  
WINTON HILL BUSINESS CENTER - BOX 161  
6110 CENTER HILL AVENUE  
CINCINNATI, OH 45224

EXAMINER
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BROWN, JENNINE M

ART UNIT	PAPER NUMBER
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1755

DATE MAILED: 06/21/2006

Please find below and/or attached an Office communication concerning this application or proceeding.

## Office Action Summary

Application No.

09/831,198

Applicant(s)

HILER II, GEORGE DOUGLAS

Examiner

Jennine M. Brown

Art Unit

1755

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

### Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

### Status

- 1) ☐ Responsive to communication(s) filed on \_\_\_\_.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

### Disposition of Claims

- 4) ☒ Claim(s) 13-33 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 13-33 is/are rejected.
- 7) ☐ Claim(s) \_\_\_\_ is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_ are subject to restriction and/or election requirement.

### Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

### Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some \* c) ☐ None of:
- ☐ Certified copies of the priority documents have been received.
  - ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_.
  - ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

### Attachment(s)

- |  |   |
|--|---|
| 1) <input checked="" type="checkbox"/> Notice of References Cited (PTO-892)  | 4) <input type="checkbox"/> Interview Summary (PTO-413)<br>Paper No(s)/Mail Date. ____. |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)   | 5) <input type="checkbox"/> Notice of Informal Patent Application (PTO-152)             |
| 3) <input checked="" type="checkbox"/> Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08)<br>Paper No(s)/Mail Date <u>7/23/01</u> . | 6) <input type="checkbox"/> Other: ____.  |

***Information Disclosure Statement***

The information disclosure statement (IDS) submitted on 7/23/2001 was considered by the examiner.

***Claim Rejections - 35 USC § 103***

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

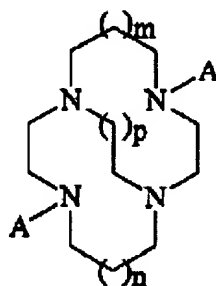
1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

Claims 13-33 are rejected under 35 U.S.C. 103(a) as being unpatentable over Busch, et al. (WO 98/39098 A1).

Busch, et al. disclose a method of making a tetraaza macrocyclic ligand having the following formula:

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To further illustrate, a preferred sub-group of the inventive transition-metal complexes includes the Mn(II), Fe(II) and Cr(III) complexes of the ligand 1.2:



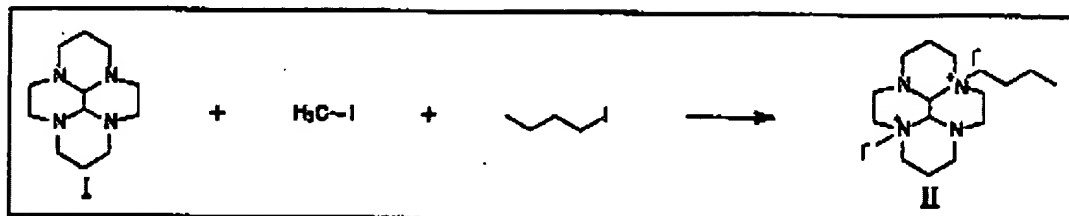
1.2

wherein  $m$  and  $n$  are integers from 0 to 2,  $p$  is an integer from 1 to 6, preferably  $m$  and  $n$  are both 0 or both 1 (preferably both 1), or  $m$  is 0 and  $n$  is at least 1; and  $p$  is 1;

and  $A$  is a nonhydrogen moiety preferably having no aromatic content; more particularly each  $A$  can vary independently and is preferably selected from methyl, ethyl, propyl, isopropyl, butyl, isobutyl, tert-butyl, C5-C20 alkyl, and one, but not both, of the  $A$  moieties is benzyl, and combinations thereof. In one such complex, one  $A$  is methyl and one  $A$  is benzyl.

where a similar synthesis is disclosed:

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(a) C<sub>4</sub>-Bicyclam Synthesis

Tetracyclic adduct I is prepared by the literature method of H. Yamamoto and K. Maruoka, *J. Amer. Chem. Soc.* (1981) **103**, 4194. I (3.00 g., 13.5 mmol) is dissolved in dry CH<sub>3</sub>CN (50 mL, distilled from CaH<sub>2</sub>). 1-Iodobutane (24.84 g., 135 mmol) is added to the stirred solution under Ar. The solution is stirred at room temperature for 5 days. 4-Iodobutane (12.42 g., 67.5 mmol) is added and the solution is stirred an additional 5 days at RT. Under these conditions, I is fully mono-alkylated with 1-iodobutane as shown by <sup>13</sup>C-NMR. Methyl iodide (26.5 g, 187 mmol) is added and the solution is stirred at room temperature for an additional 5 days. The reaction is filtered using Whatman #4 paper and vacuum filtration. A white solid, II, is collected (6.05 g., 82%).

<sup>13</sup>C NMR (CDCl<sub>3</sub>) 16.3, 21.3, 21.6, 22.5, 25.8, 49.2, 49.4, 50.1, 51.4, 52.6, 53.9, 54.1, 62.3, 63.5, 67.9, 79.1, 79.2 ppm. Electro spray Mass Spec. (MH<sup>+</sup>/2, 147).

II (6.00 g., 11.0 mmol) is dissolved in 95% ethanol (500 mL). Sodium borohydride (11.0 g., 290 mmol) is added and the reaction turns milky white. The reaction is stirred under Ar for three days. Hydrochloric acid (100 mL, concentrated) is slowly dripped into the reaction mixture over 1 hour. The reaction mixture is evaporated to dryness using a rotoevaporator. The white residue is dissolved in sodium hydroxide (500 mL, 1.00N). This solution is extracted with toluene (2 x 150 mL). The toluene layers are combined and dried with sodium sulfate. After removal of the sodium sulfate using filtration, the toluene is evaporated to dryness using a rotoevaporator. The resulting oil is dried at room temperature under high vacuum (0.05 mm) overnight. A colorless oil results 2.95 g., 90%. This oil (2.10 g.) is distilled using a short path distillation apparatus (still head temperature 115 C at 0.05 mm). Yield: 2.00 g. <sup>13</sup>C NMR (CDCl<sub>3</sub>) 14.0, 20.6, 27.2, 27.7, 30.5, 32.5, 51.2, 51.4, 54.1, 54.7, 55.1, 55.8, 56.1, 56.5, 57.9, 58.0, 59.9 ppm. Mass Spec. (MH<sup>+</sup>, 297).

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**(a) C<sub>8</sub>-Bcyclam Synthesis:**

This ligand is synthesized similarly to the C<sub>4</sub>-Bcyclam synthesis described above in Example 2(a) except that 1-iodooctane is used in place of the 1-iodobutane.

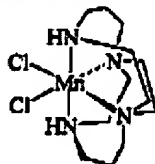
Mass Spec. (MH<sup>+</sup>, 353).

**(b) [Mn(C<sub>8</sub>-Bcyclam)Cl<sub>2</sub>] Synthesis**

This complex is made similarly to the [Mn(C<sub>4</sub>-Bcyclam)Cl<sub>2</sub>] synthesis described above in Example 2(b) except that C<sub>8</sub>-Bcyclam is used in place of the C<sub>4</sub>-Bcyclam.

Ion Spray Mass Spectroscopy shows one major peak at 452 mu corresponding to [Mn(Bg-Bcyclam)(formate)]<sup>+</sup>.

**Example 5. Synthesis of [Mn(H<sub>2</sub>-Bcyclam)Cl<sub>2</sub>] where H<sub>2</sub>-Bcyclam =**  
**1,5,8,12-tetraaza-bicyclo[6.6.2]hexadecane**



The H<sub>2</sub>-Bcyclam is synthesized similarly to the C<sub>4</sub>-Bcyclam synthesis described above except that benzyl bromide is used in place of the 1-iodobutane and the methyl iodide. The benzyl groups are removed by catalytic hydrogenation. Thus, the resulting 5,12-dibenzyl-1,5,8,12-tetraaza-bicyclo[6.6.2]hexadecane and 10% Pd on charcoal is dissolved in 85% acetic acid. This solution is stirred 3 days at room temperature under 1 atm. of hydrogen gas. The solution is filtered through a 0.2 micron filter under vacuum. After evaporation of solvent using a rotary evaporator, the product is obtained as a colorless oil. Yield: 90+%.

The Mn complex is made similarly to the [Mn(Bcyclam)Cl<sub>2</sub>] synthesis described in Example 1(b) except that the that H<sub>2</sub>-Bcyclam is used in place of the Bcyclam.

**Elemental Analysis:** %C, 40.92; %H, 7.44; %N, 15.91; theoretical for [Mn(H<sub>2</sub>-Bcyclam)Cl<sub>2</sub>], MnC<sub>12</sub>H<sub>26</sub>N<sub>4</sub>Cl<sub>2</sub>, MW = 352.2. Found: %C, 41.00; %H, 7.60; %N, 15.80. FAB+ Mass Spectroscopy shows one major peak at 317 mu corresponding to [Mn(H<sub>2</sub>-Bcyclam)Cl]<sup>+</sup> and another minor peak at 352 mu corresponding to [Mn(H<sub>2</sub>-Bcyclam)Cl<sub>2</sub>]<sup>+</sup>.

It would have been apparent to one of ordinary skill in the art to substitute the 4-iodobutane for an 2-iodoethane compound to produce a ligand with ethyl R groups, to which MnCl<sub>2</sub> would be added to produce the catalytic compound claimed. The prior art

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states that nickel compounds such as Raney nickel may be alternate embodiments to the manganese compound claimed and that the anionic ligands which charge balance the compound may be:

**optionally, one or more non-macropolycyclic ligands, preferably selected from the group consisting of  $H_2O$ ,  $ROH$ ,  $NR_3$ ,  $RCN$ ,  $OH^-$ ,  $OOH^-$ ,  $RS^-$ ,  $RO^-$ ,  $RCOO^-$ ,  $OCN^-$ ,  $SCN^-$ ,  $N_3^-$ ,  $CN^-$ ,  $F^-$ ,  $Cl^-$ ,  $Br^-$ ,  $I^-$ ,  $O_2^-$ ,  $NO_3^-$ ,  $NO_2^-$ ,  $SO_4^{2-}$ ,  $SO_3^{2-}$ ,  $PO_4^{3-}$ , organic phosphates, organic phosphonates, organic sulfates, organic sulfonates, and aromatic N donors such as pyridines, pyrazines, pyrazoles, imidazoles, benzimidazoles, pyrimidines, triazoles and thiazoles with R being H, optionally substituted alkyl, optionally substituted aryl;**

therefore it would have been in the purveyance of one in the art to have modified the compounds and methods used in the claimed method to produce a desired catalyst based on number of carbons in the tetraaza compound preferred, based on the substituents on the nitrogen group, based on the catalytic metal desired and the charge balancing anion for the metal center.

A prima facie case of obviousness may be made when chemical compounds have very close structural similarities and similar utilities. "An obviousness rejection based on similarity in chemical structure and function entails the motivation of one skilled in the art to make a claimed compound, in the expectation that compounds similar in structure will have similar properties." In re Payne, 606 F.2d 303, 313, 203 USPQ 245, 254 (CCPA 1979). See In re Papesch, 315 F.2d 381, 137 USPQ 43 (CCPA 1963) (discussed in more detail below) and In re Dillon, 919 F.2d 688, 16 USPQ2d 1897 (Fed. Cir. 1991) (discussed below and in MPEP § 2144) for an extensive review of the case

law pertaining to obviousness based on close structural similarity of chemical compounds. See also MPEP § 2144.08, paragraph II.A.4.(c).

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Jennine M. Brown whose telephone number is (571) 272-1364. The examiner can normally be reached on M-R 9:30 AM - 7:30 PM; Fridays off.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jerry Lorengo can be reached on (571) 272-1233. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

jmb

  
J.A. LORENGO  
SUPERVISORY PATENT EXAMINER